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Iodine Sorption Study on the Proposed Use of Viton A In A Shuttle Galley Water Accumulator

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ABSTRACT

The installation of a Viton A accumulator in the Shuttle galley has been proposed to prevent over-pressurization of the hot water supply system. A laboratory study was conducted to determine if there would be any interaction between the Viton A material and the iodine used to disinfect the water. Coupons of Viton A were exposed for 24 hours to aqueous iodine solutions similar in quality to the Shuttle's potable water. Changes in the iodine residual were monitored to determine the rate of iodine sorption by the coupon. Total organic carbon (TOC) was monitored to determine the rate of desorption of organic materials from the Viton A. The same coupons were then soaked in reagent-grade water for 24 hours, and iodine was monitored to determine the rate of iodine desorption. The coupons were again exposed to iodine solutions for 24 hours and iodine and TOC were monitored. No significant change in the iodine sorption rate was detected between the first and second exposures. A triangle taste test indicated at a 0.1% confidence level that the water exposed to Viton A had a different taste which was less acceptable to the panelists.

INTRODUCTION

The Shuttle's potable water system consists of four parallel water tanks replenished continuously with fuel cell water. To disinfect the water, it is passed through an iodinated ion exchange resin. Before its use in the Shuttle galley, the iodinated water is heated or chilled. To prevent overpressurization of the galley hot water supply system, the addition of an accumulator to provide expansion volume is being considered. Because of the high cost and amount of time involved in obtaining stainless steel bellows tanks, the use of an accumulator consisting of a Viton A diaphragm has been proposed.

Viton A is an elastomeric copolymer of vinylidine fluoride and hexafluoro propylene produced by E. I. duPont de Nemours. Experience with elastomers in iodinated water systems has shown that they may sorb iodine and reduce the biocide residual in the water, creating the opportunity for microbial proliferation. As a result, some concern has been raised about the effect of Viton A on the quality of the Shuttle's water supply. If the desorption of iodine back into the water could be confirmed and quantitated, the concern about microbial growth would be lessened because a biocide residual would be maintained. Another concern with the use of Viton A is that elastomers are also known to release organic compounds into exposed water, thus affecting the water's palatability and potability.

To investigate what effect Viton A may have on water quality, a study was conducted in the NASA/ Johnson Space Center (JSC) Water and Food Analytical Laboratory to determine to what extent iodine interacts with Viton A. The specific objectives of the study were to determine the following:

1. The extent and rate of iodine sorption onto a given surface area of Viton A from an iodine solution similar to the Space Shuttle's water supply

2. The extent and rate of iodine desorption into water from the iodine-exposed Viton A

3. The rate of organic carbon leaching from Viton A into iodinated water

4. If any taste is imparted to iodine solutions due to exposure to Viton A

MATERIALS AND METHODS

Solution volumes and Viton A coupon sizes used for this study were chosen so that changes in iodine and TOC levels could be detected and rates of change calculated. Solutions were sampled periodically over a 24-hour period and analyzed for iodine and TOC. Leaching of sorbed iodine was tested by removing the coupons from the iodine solutions and placing them for 24 hours in bottles containing 200 ml of reagent-grade water. Coupons were re-exposed to the same concentration of iodine for another 24-hour period. Iodine sorption and TOC changes were again monitored. After exposure testing, the solutions were composited and submitted for taste testing.

Materials

Coupons.- Viton A samples were obtained through Lockheed (LEMSCO) from Greer Hydraulics, the manufacturer of the proposed accumulator. The samples were cleaned by the JSC Technical Services Division, according to Level 300 flight hardware specifications.[1] Six coupons (four at 1 inch by 2 inches and two at 1.5 inches by 3 inches) were then cut with a razor blade from the central portion of the Viton A sample. Desired coupon dimensions were outlined on the packaging of the cleaned Viton A samples and the sample was cut along these lines. To determine the parameters for coupon surface area estimation from coupon weight, a sample coupon was weighed on an analytical balance and its external dimensions were measured. The Viton A coupon selected for the surface area standard weighed 4.8917 g. Its dimensions were 5.305 cm by 2.538 cm by 0.190 cm giving an area of 29.91 sq cm, which corresponds to 6.114 sq cm/g. This ratio was used to approximate the surface area from the weight of each coupon (table I). The remaining coupons were used for the exposure testing.

Water.- Reagent-grade water was prepared using distilled, deionized water passed through a Milli-Q Plus ® water polishing system. Quality control analyses indicated 18 megohms-cm resistivity, 20 ppb TOC, and 11 CFU/100 ml.

Bottles.- Six 500-ml reagent bottles were cleaned with 0.5 g/ml sodium hydroxide solution and rinsed 10 times with approximately 200 ml of reagent-grade water. The bottles were then wrapped in aluminum foil to minimize exposure to light and labeled as described in table II.

Test Procedures

First Iodine Exposure.- Exposure solutions were composed of a 1:1 mole ratio of iodine/iodide to simulate Space Shuttle potable water. A stock solution was prepared by adding 15 ml of stock potassium iodide solution (1000 ppm iodide) and 52.6 ml of iodine saturated water (approximately 290 ppm iodine) to a 500-ml volumetric flask and diluting to the mark. The resulting solution was estimated to be 30 ppm iodine. The exact concentration was not considered critical since the final exposure solutions were to be analyzed for iodine residual. Each 500-ml exposure bottle was then tared, and a volume of stock iodine solution added as indicated in table II. The final volume of 300 ml was obtained by adding reagent-grade water until a final solution weight of 300 g was reached.

The coupons were weighed on an analytical balance and placed in the appropriate reagent bottles as described in table II. Initial samples were taken at this time and analyzed for iodine and TOC. The exposure bottles were then sealed with glass stoppers and placed on an agitator which consisted of a tray mounted on an intentionally unbalanced magnetic stirrer to generate a vibration. Bottles were swirled by hand before each sample was taken and periodically between samplings. Subsequent 10-ml samples were taken at 0.25 hr, 0.5 hr, 1 hr, 4 hr, and 24 hr for iodine analysis. Twenty-milliliter samples were taken at 4 hr and 24 hr for TOC analysis. Iodine samples were withdrawn by pipette, dispensed directly into a 50-ml volumetric flask, and immediately analyzed. TOC samples were transferred by pipette to a glass sample vial and analyzed as soon as possible.

Iodine Desorption.- Six 250-ml glass reagent bottles were cleaned and labeled using the same method as described for the exposure bottles. The six bottles were then filled with 200 ml of reagent-grade water. After the first exposure period, the coupons were removed from the exposure bottles, rinsed with a minimum amount of reagent-grade water, and placed into the appropriately labeled 250-ml bottle. The rinse water was saved and later analyzed for iodine. The same sampling sequence as used in the iodine exposure test was then followed by drawing 10.0 ml for iodine analysis only.

Second Iodine Exposure.- After 24 hours of leaching, the coupons were removed from the 250-ml bottles (saving the leachate) and rinsed with reagent-grade water. They were then placed into the 500-ml iodine exposure bottles which had been cleaned and prepared with fresh iodine solutions. The original exposure and sampling process was repeated. The coupons were left in the exposure solutions beyond the 24-hour period to obtain some long-term data on iodine depletion and TOC leaching.

Sample Analysis

Iodine Analysis.- Iodine concentrations were determined using the leuco crystal violet (LCV) method.[2] To minimize changes in the solution volumes, 10-ml samples were taken instead of 25 ml. Some of the sensitivity that was lost due to the small sample size was regained by developing the LCV color in a 50-ml volumetric flask, rather than in a 100-ml flask as called for in the reference. The developing reagent volumes were halved accordingly. Absorbances were determined with a Shimadzu model UV-265 spectrophotometer set at a wavelength of 592 nm. A 1-cm quartz cell was used for the exposure solutions. Using this pathlength, the minimum iodine detectability was determined to be approximately 0.5 ppm since at this concentration the absorbance values approached 0.000 absorbance units (fig. 1). To increase sensitivity, a 10-cm quartz cell was used for the leachate.

TOC Analysis.- TOC analysis was performed using the Wet Oxidation Method [2] with an O.I. Corporation Model 700 Carbon Analyzer.

Taste Testing.- To determine if any unpleasant taste had been imparted to the sample from the Viton A, a taste test was coordinated through the JSC Man-Systems Division food group. Fourteen panelists participated. The test was conducted according to the ASTM Manual on Sensory Testing Methods.[3] The iodine concentration in the composite sample was calculated from the average of the final exposure solution concentrations as measured by the LCV test. A blank solution of similar composition was prepared for use in the taste test. A triangle test was used to detect a taste difference. Each panelist was presented with three samples, two alike and one odd. They were asked to identify the odd sample. Using the forced-choice method, the palatability of the sample was compared to the control in conjuction with the triangle test.[3] Each panelist was required to choose the sample they preferred and to indicate the degree of difference from the other samples.

RESULTS

Iodine Sorption

Results of the iodine analysis from the exposure experiments are listed in table III. The iodine blank (B2) was used to determine the precision of the LCV test and showed standard deviations for the first and second iodine exposure series of 0.156 ppm and 0.057 ppm, respectively, over the first 4 hours of the test. Unfortunately, precise determination of the small iodine depletion during this exposure period was not possible using only a 10.0 ml sample aliquot. Measurable iodine depletions were observed after 4 hours as illustrated in figures 2 and 3. The slopes indicate the rate of iodine sorption in ppm/hr during the experiment. The relationship to coupon area is seen in the different slopes of lines for the small and large coupons. The large coupons show a larger negative slope.

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To determine the rate constants for the sorption process, the rate order was determined first. The iodine concentration data were fitted to zero-, first-, and second-order rate laws using the integrated rate equations.[4] The results are shown in table IV. The linear correlation (R) values indicate that the sorption is probably not zero order. The fit was generally better to the first-order expression than to the second-order. A plot of the test data confirmed that the first-order expression gave the best fit. The integrated first-order rate equation is as follows:

$$[A]_t = [A]_0 \exp(-K_1 t)$$

A good fit to a rate equation allows calculation of reagent concentration at any time (t) after a process has started given the initial concentration ($[A]_0$). The rate constants (K_1) for each coupon were determined from the slope of the plot $\ln([A]_0/[A]_t)$ versus t for the first-order test. The values are listed in table V along with the values after normalization to the coupon area. The large coupons exhibited a slower rate of sorption per unit area than the small coupons. This is indicated by the larger first-order rate constants. Sample collection was concentrated in the first 4 hours of exposure because previous experience indicated that equilibrium may be reached quickly. The data show, however, that a better rate-expression fit may have been achieved if the samplings had been more evenly distributed over the 24-hour exposure period.

Iodine Desorption

Iodine levels in the desorption samples and coupon rinses were below detection limits indicating insignificant iodine leaching. Thus, the iodine sorbed during the first exposure was not readily desorbed from the Viton A. This may be due to a high affinity of Viton A to iodine or a chemical change in the iodine, such as reduction to iodide or conversion to an iodo-organic, resulting in no detectable iodine residual.

TOC Analysis

TOC analysis results are listed in table VI. The results of the first exposure were not included because of a malfunction in the reagent delivery system on the OIC instrument. Therefore, the TOC leaching rates are based on the second exposure only. Consistent instrument performance for the TOC analyses were assured by periodically monitoring a 1.0-ppm standard with the sample series. No significant changes in the standard analyses were observed during the second-exposure series.

The rate order was determined using the same procedure employed for the iodine sorption process for TOC leaching during the first 24 hours of exposure. The rate constants and R values are listed in table VII. The best fit was found for the zero-order rate law, $[A]_t = K_0 t$. The rate constants (K_0) are listed in table VIII along with the values after normalization to the coupon area.

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<u>Taste Test</u>

Samples 1-4 from the first iodine exposure wee composited and used to determine if exposure to Viton A adversely affected the taste of the water. Twelve of the 14 panelists correctly identified the odd sample whether it was the sample or the control. To a 0.1% confidence interval, this indicates that there is a taste difference. Of the 12 that correctly identified the odd sample, 10 found the difference between the samples to be moderate to extreme, 10 preferred the blank over the Viton A-exposed water, 1 preferred the exposed sample, and 1 had no preference.

DISCUSSION

The data indicate that iodine affects the TOC levels in the Viton A-exposed solutions (table VI). The coupon blank (B1) is a control against which the TOC leaching due to the iodinated exposure solutions may be compared. In the blank sample, the TOC level would normally be expected to rise at a rate proportional to the size of the coupons and remain constant at some equilibrium point. Inspection of the TOC data shows that this did occur. In the iodinated exposure solutions, however, initial TOC leaching rates were accelerated. In addition, after continued exposure, TOC concentrations decreased in three of the four iodinated exposure solutions. This indicates an interaction of the iodine residual not only with the Viton A, but also with the leached organic material.

The reduction in TOC could be caused by microbial carbon consumption resulting in CO_2 production, iodine interference in TOC analysis, formation of volatile organics which are not detected in TOC analysis, or resorption of organics by the coupon. The first of these possibilities was ruled out because a similar reduction in TOC would have been observed in the coupon blank if there were any microbial activity. Further evidence for this conclusion was obtained by inspecting the inorganic carbon levels in the samples. If microorganisms were active, an increase in inorganic carbon due to microbial CO_2 production would be expected. No increase was observed.

The possibility of errors in the TOC analysis resulting from the presence of iodine in the samples was investigated and also ruled out. A 2-ppm TOC standard was analyzed for TOC and spiked with 5 ml of saturated iodine solution, resulting in a 3-ppm iodine solution. The sample was again analyzed and no difference, other than the slight dilution of the standard, was observed. The standard was again spiked with iodine, resulting in approximately a 5-ppm iodine solution, and TOC results remained unaffected.

Both volatile organic formation and organic resorption by a coupon involve the reaction of aqueous iodine with the Viton A coupons or the aqueous organic material leached from them. These mechanisms would result in the formation of an organic compound with different physical properties than the compounds originally leached from the Viton A. This is supported by the fact that the reduction in TOC was seen in the iodinated solutions, but not in the coupon blank.

The formation of iodinated organics could also result in an iodine demand in the solution after exposure to the coupon. To evaluate this possibility, the remainder of the first-exposure composite used in the taste test was retested for iodine. The composite had been refrigerated and stored in a glass reagent bottle covered with aluminum foil. Extrapolation of the iodine blank data indicates

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that without an introduced iodine demand there should still be a measurable iodine residual after 10 days. Ten days after the initial exposure began the iodine residual had been almost eliminated in the absence of Viton A coupons.

Assuming that iodine is reacting with organics leached from the Viton A, neither of the above mechanisms can be ruled out based on available data. The first mechanism involves the formation of volatile organic compounds which are not detected by the TOC analysis procedure used in the experiment. The compounds would be lost in the first stage of analysis in which inorganic carbon and volatile materials are driven from the sample by the carrier gas. This pathway may be verified by purgeable organic carbon (POC) analysis. An increase in POC would indicate formation of a more volatile organic species that is not normally detectable in TOC analysis.

The mechanism involving resorption of organics by Viton A can be verified either by showing no increase in POC, thus eliminating that mechanism of TOC depletion, or by gas chromatograph analysis of the water samples before and after exposure to detect any change in the identity of the organic contaminants.

CONCLUSIONS

The results of this study show that Viton A demonstrates a definite demand for iodine. Iodine lost from solution during exposure is not readily recoverable from the Viton A. A change in the iodine depletion rate due to previous exposure to iodine was less than could be confidently measured with the LCV procedure.

It was also determined that the Viton A leached a significant amount of organic carbon during exposure to iodinated water. A measurable TOC decrease in the iodinated solutions suggests that the iodine biocide interacts with the leached organics resulting in new organic compounds with different physical properties (i.e., volatilization or sorbability). The toxicological properties of these new organic compounds are unknown. Because of the proximity of the proposed accumulator to the galley hot water supply sytem, the interaction between the Viton A and iodine biocide could be increased by the associated heat.

Using the triangle taste test, it was also determined within a 0.1% confidence interval that exposure of the iodinated water to Viton A negatively altered the taste of the water. No definite conclusion may be drawn from this study about the effect of the proposed accumulator on the taste of the galley water under normal operating conditions since the accumulator volume is not flushed. If cycling into the accumulator were to occur, however, the palatability of the water would be adversely affected by the Viton A diaphragm.

Given the exposure area at the interface between the proposed accumulator and the water distribution system, and considering the volume of the STS water system, the use of a Viton A diaphragm does not appear to pose a serious threat to water quality by decreasing the iodine residual of the entire system. A problem could arise, however, because the proposed accumulator design includes an entrapped volume of water. This water would come into contact with a large surface area of Viton A which is not actively flushed with iodinated water. The large Viton A surface area within

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the accumulator would deplete the biocide residual in the entrapped water in a matter of hours, creating an environment suitable for microbial proliferation. The release of organic carbon from the diaphragm could also encourage microbial growth. Such microbial activity would be a source of toxins and pathogenic microorganisms.

From the results of this study, Viton A cannot be recommended for use in the proposed galley accumulator because of the adverse affects to water quality resulting from exposure to Viton A. This position could be reconsidered if further study can demonstrate that pretreatment of Viton A eliminates the organic leaching, the iodine demand, and the adverse effect on palatability. It is recommended that such studies be conducted if the Viton A material continues to be considered for use.

REFERENCES

1. Contamination Control Requirements Manual: JSCM 5322, Revision B. Lyndon B. Johnson Space Center (Houston, Texas), November 1982, pp. a12, 13.

2. Greenberg, Arnold E., et al., eds.: Standard Methods for the Examination of Water and Wastewater, 16th edition. American Public Health Association (Washington, D.C.), 1982, pp. 513-515, 365-366.

3. American Society for Testing Materials: Manual on Sensory Testing Methods. ASTM Special Technical Publication 434, (Philadelphia, Pennsylvania), 1985, pp. 24-29.

4. Atkins, P. W.: Physical Chemistry, 2nd edition. W. H. Freeman and Co., 1982, pp. 924-932.

TABLE I.- COUPON DATA

Coupon	Mass (g)	Area (sq cm)
B1	4.9268	30.1
1	4.9941	30.53
2	10.6424	65.07
3	4.9944	30.54
4	10.6899	65.36

TABLE II.- IODINE EXPOSURE TEST SET UP

Bottle ID	Coupon dimensions (in × in)	Volume of stock I2/I - added (ml)	Approx. I2 conc. (ppm)
B1	1 × 2	0	0
B2	---	40	4
1	1 × 2	20	2
2	1.5 × 3	20	2
3	1 × 2	40	4
4	1.5 × 3	40	4

TABLE III.- IODINE CONCENTRATIONS (PPM) DURING VITON A EXPOSURES

Time (hr)	Bottle ID/Coupon Size					
	B1 (1″ × 2″)	B2 (------)	1 (1″ × 2″)	2 (1.5″ × 3″)	3 (1″ × 2″)	4 (1.5″ × 3″)
			Exposure 1			
0.0	<0.5	6.3[a]	3.3	3.2	6.6	4.4[a]
0.25	<0.5	6.6	3.3	3.1	6.4	6.0
0.5	<0.5	6.7	3.3	3.2	6.5	6.4
1.0	<0.5	6.5	3.3	3.1	6.4	6.1
4.0	<0.5	6.4	3.1	2.6	6.0	5.6
24.0	<0.5	6.1	2.1	1.0	4.8	3.0
			Exposure 2			
0.0	<0.5	5.9	2.8	2.8	6.1	5.9
0.25	<0.5	5.8	2.8	2.8	5.9	5.3[a]
0.5	<0.5	5.9	2.8	2.8	5.9	6.0
1.0	<0.5	5.8	2.8	2.7	5.9	4.8[a]
4.0	<0.5	5.8	2.6	2.5	5.7	5.2
24.0	<0.5	5.5	1.8	1.0	4.5	3.3
72.0	<0.5	5.6	0.7	<0.5	<0.5	0.7

[a]Data rejected because deviation from mean first-hour values of similarly prepared solutions exceeded the calculated standard deviation of the test.

TABLE IV.- IODINE SORPTION RATE TEST DATA

Order	Parameter	Coupon ID/Coupon Size			
		1 ($1'' \times 2''$)	2 ($1.5'' \times 3''$)	3 ($1'' \times 2''$)	4 ($1.5'' \times 3''$)
		Exposure 1			
Zero	Ko	0.051	0.091	0.071	0.135
	R	0.9993	0.9933	0.9869	0.9933
First	K1	0.044	0.112	0.029	0.71
	R	0.9993	0.9993	0.9916	0.9966
Second	K2	0.0019	0.0073	0.0006	0.0019
	R	0.9986	0.9981	0.9950	0.9969
		Exposure 2			
Zero	Ko	0.042	0.075	0.062	0.109
	R	0.9981	0.9996	0.9935	0.9902
First	K1	0.044	0.1000	0.027	0.068
	R	0.9992	0.9986	0.9955	0.9964
Second	K2	0.0021	0.0069	0.0006	0.0014
	R	0.9993	0.9957	0.9967	0.9991

TABLE V.- IODINE SORPTION RATE CONSTANTS

Coupon ID/ Size (in × in)	Approximate initial [I] (ppm)	Iodine sorption rate constant K1		Iodine sorption rate constants normalized to coupon area	
		Exp 1	Exp 2	Exp 1	Exp 2
1/ (1 × 2)	3	0.044	0.044	1.4 E-3	1.4 E-3
2/ (1.5 × 3)	3	0.112	0.100	1.7 E-3	1.5 E-3
3/ (1 × 2)	6	0.029	0.027	0.9 E-3	0.9 E-3
4/ (1.5 × 3)	6	0.071	0.068	1.1 E-3	1.0 E-3

TABLE VI.- TOC ANALYSIS RESULTS (PPM)

	Bottle ID/Coupon Size					
Time	B1 (1″ × 2″)	B2 (------)	1 (1″ × 2″)	2 (1.5″ × 3″)	3 (1″ × 2″)	4 (1.5″ × 3″)
	Exposure 2					
0.0 hr	0.22	0.23	0.31	0.57	0.47	0.44
4.0 hr	0.58	0.34	0.64	1.29	0.71	1.15
24.0 hr	2.70	0.41	3.17	4.54	3.39	4.60
3 wks	4.65	0.60	3.94	2.52	3.24	2.27

TABLE VII.- TOC CONCENTRATION (PPM) LEACHING RATE TEST DATA

		Coupon ID/Coupon Size			
Order	Parameter	1 (1″ × 2″)	2 (1.5″ × 3″)	3 (1″ × 2″)	4 (1.5″ × 3″)
		Exposure 2			
Zero	K_o	0.122	0.165	0.126	0.173
	R	0.9987	0.9999	0.9966	0.9999
First	K_1	0.211	0.182	0.187	0.204
	R	0.9883	0.9701	0.9991	0.9655
Second	K_2	-0.026	-0.013	-0.018	-0.017
	R	-0.9005	-0.8649	-0.9704	-0.8390

TABLE VIII.- CARBON LEACHING RATE CONSTANTS

Coupon ID/ Size (in × in)	Approximate initial [I2] (ppm)	Carbon leaching rate constant Ko (L/mg-hr)	Carbon leaching rate constants normalized to coupon area (L/mg-hr-cm³)
		Exposure 2	
B1 (1 × 2)	0	0.104	3.5 E-3
1 (1 × 2)	3	0.122	4.0 E-3
2 (1.5 × 3)	3	0.165	2.5 E-3
3 (1 × 2)	6	0.126	4.1 E-3
4 (1.5 × 3)	6	0.173	2.6 E-3

Figure 1.- Calibration curve for iodine analysis by the leuco crystal violet method using a 1.0 cm pathlength at 592 nm.
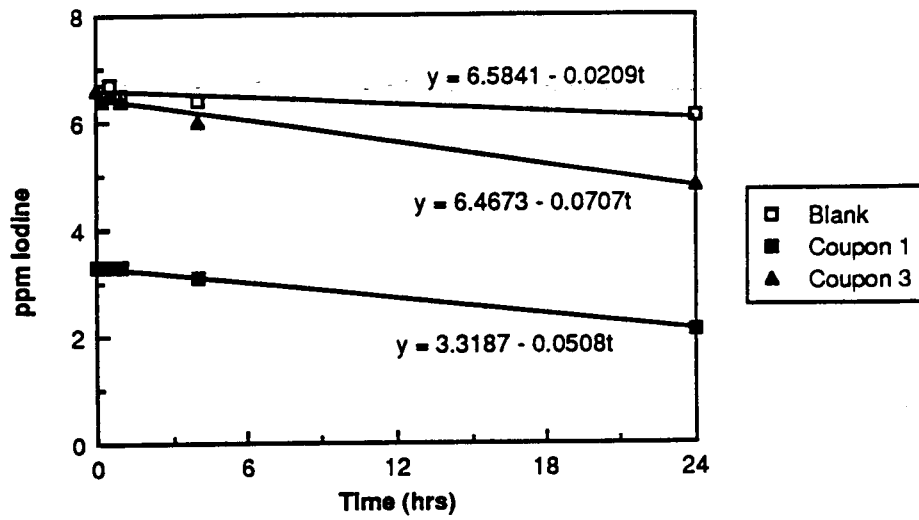
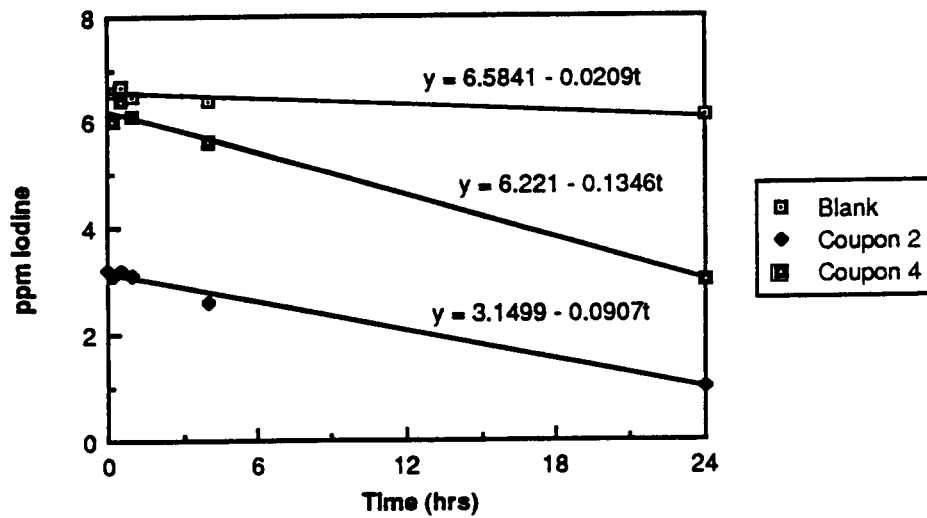Figure 2a.- Iodine sorption by Viton A. First exposure with 1″ × 2″ coupons.

Figure 2b.- Iodine sorption by Viton A. First exposure with 1.5″ × 3″ coupons.
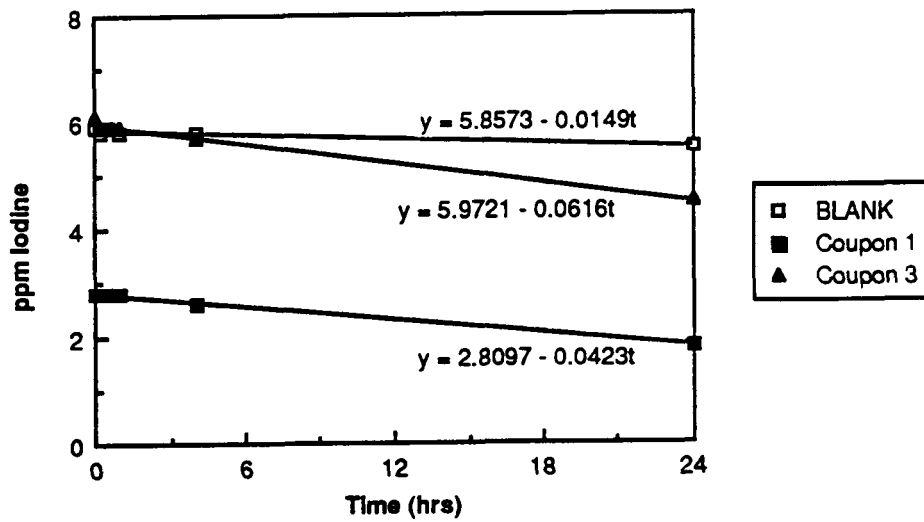
13

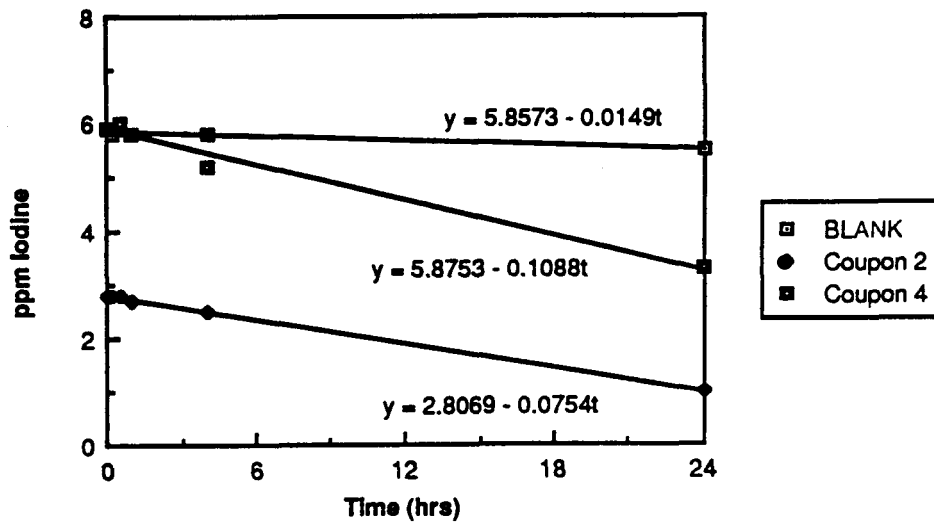Figure 3a.- Iodine sorption by Viton A. Second exposure with 1" × 2" coupons.

Figure 3b.- Iodine sorption by Viton A. Second exposure with 1.5" × 3" coupons.

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16. Abstract

The installation of a Viton A accumulator in the Shuttle galley has been proposed to prevent overpressurization of the hot water supply system. A laboratory study was conducted to determine if there would be any interaction between the Viton A material and the iodine used to disinfect the water. Coupons of Viton A were exposed for 24 hours to aqueous iodine solutions similar in quality to the Shuttle's potable water. Changes in the iodine residual were monitored to determine the rate of iodine sorption by the coupon. Total organic carbon (TOC) was monitored to determine the rate of desorption of organic materials from the Viton A. The same coupons were then soaked in reagent-grade water for 24 hours, and iodine was monitored to determine the rate of iodine desorption. The coupons were again exposed to iodine solutions for 24 hours and iodine and TOC were monitored. No significant change in the iodine sorption rate was detected between the first and second exposures. A triangle taste test indicated at a 0.1% confidence level that the water exposed to Viton A had a different taste which was less acceptable to the panelists.

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